

FORM PTO-1390 (Modified)  
(REV 11-2000)

U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE

ATTORNEY DOCKET NUMBER

**TRANSMITTAL LETTER TO THE UNITED STATES  
DESIGNATED/ELECTED OFFICE (DO/EO/US)  
CONCERNING A FILING UNDER 35 U.S.C. 371**

221111US0PCT

U.S. APPLICATION NO. (IF KNOWN, SEE 37 CFR

10/089099

INTERNATIONAL APPLICATION NO.  
PCT/FR00/02841

INTERNATIONAL FILING DATE  
12 October 2000

PRIORITY DATE CLAIMED  
13 October 1999

TITLE OF INVENTION

**BORON-BASED CONFINEMENT MATRIX FOR THE STORAGE OR INCINERATION OF LONG-LIFE  
RADIOACTIVE ELEMENTS**

APPLICANT(S) FOR DO/EO/US

**Sylvain DEUTSCH et al.**

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.
2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
3. ☒ This is an express request to begin national examination procedures (35 U.S.C. 371(f)). The submission must include items (5), (6), (9) and (24) indicated below.
4. ☒ The US has been elected by the expiration of 19 months from the priority date (Article 31).
5. ☒ A copy of the International Application as filed (35 U.S.C. 371 (c) (2))
  - a. ☐ is attached hereto (required only if not communicated by the International Bureau).
  - b. ☒ has been communicated by the International Bureau.
  - c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US).
6. ☒ An English language translation of the International Application as filed (35 U.S.C. 371(c)(2)).
  - a. ☒ is attached hereto.
  - b. ☐ has been previously submitted under 35 U.S.C. 154(d)(4).
7. ☒ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371 (c)(3))
  - a. ☐ are attached hereto (required only if not communicated by the International Bureau).
  - b. ☐ have been communicated by the International Bureau.
  - c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
  - d. ☒ have not been made and will not be made.
8. ☐ An English language translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).
9. ☐ An oath or declaration of the inventor(s) (35 U.S.C. 371 (c)(4)).
10. ☐ An English language translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371 (c)(5)).
11. ☐ A copy of the International Preliminary Examination Report (PCT/IPEA/409).
12. ☒ A copy of the International Search Report (PCT/ISA/210).

**Items 13 to 20 below concern document(s) or information included:**

13. ☐ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
14. ☐ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
15. ☒ A **FIRST** preliminary amendment.
16. ☐ A **SECOND** or **SUBSEQUENT** preliminary amendment.
17. ☐ A substitute specification.
18. ☐ A change of power of attorney and/or address letter.
19. ☐ A computer-readable form of the sequence listing in accordance with PCT Rule 13ter.2 and 35 U.S.C. 1.821 - 1.825.
20. ☐ A second copy of the published international application under 35 U.S.C. 154(d)(4).
21. ☐ A second copy of the English language translation of the international application under 35 U.S.C. 154(d)(4).
22. ☐ Certificate of Mailing by Express Mail
23. ☒ Other items or information:

**Notice of Priority/ PCT/IB/304/ PCT/IB/308**

**Request for Consideration of Documents Cited in International Search Report**

ATTORNEY'S DOCKET NUMBER  
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**Notice of Priority/ PCT/IB/304/ PCT/IB/308**

**Request for Consideration of Documents Cited in International Search Report**



221111US-0 PCT

IN THE UNITED STATES PATENT & TRADEMARK OFFICE

IN RE APPLICATION OF: :  
SYLVAIN DEUTSCH ET AL : ATTN: APPLICATION DIVISION  
SERIAL NO: NEW U.S. PCT APPLN :  
(BASED ON PCT/FR00/02841)  
FILED: HEREWITH :  
FOR: BORON-BASED CONFINEMENT  
MATRIX FOR THE STORAGE OR  
INCINERATION OF LONG-LIFE  
RADIOACTIVE ELEMENTS

PRELIMINARY AMENDMENT

ASSISTANT COMMISSIONER FOR PATENTS  
WASHINGTON, D.C. 20231

SIR:

Prior to examination on the merits, please amend the above-identified application as follows.

IN THE CLAIMS

Please amend the claims as shown in the marked-up copy following this amendment to read as follows.

7. (Amended) Confinement matrix according to claim 1 for the incineration of at least one radioactive element, wherein the boron of the boron compound is enriched with <sup>11</sup>B.

10. (Amended) Method according to claim 8, wherein the powder mixture also comprises one or more additives chosen from metals, catalysts, metal oxides or the adjuvants required to form the matrix or improve its properties.

11. (Amended) Method according to claim 8, wherein the boron precursor is chosen from  $B_2O_3$ ,  $H_3BO_3$ ,  $B_3Si$ ,  $B_6O$  and  $B_4C$ .

REMARKS

Claims 1-15 are active in the present application. Claims 7 and 10-11 have been amended to remove multiple dependencies. No new matter is added. An action on the merits and allowance of claims is solicited.

Respectfully submitted,

OBLON, SPIVAK, McCLELLAND,  
MAIER & NEUSTADT, P.C.

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<b>Marked-Up Copy</b>
Serial No:
Amendment Filed on:
4-12-2002

IN THE CLAIMS

--7. (Amended) Confinement matrix according to [any of claims 1 to 6] claim 1 for the incineration of at least one radioactive element, wherein the boron of the boron compound is enriched with  $^{11}\text{B}$ .

10. (Amended) Method according to [any of claims 8 and 9] claim 8, wherein the powder mixture also comprises one or more additives chosen from metals, catalysts, metal oxides or the adjuvants required to form the matrix or improve its properties.

11. (Amended) Method according to [any of claims 8 to 10] claim 8, wherein the boron precursor is chosen from  $\text{B}_2\text{O}_3$ ,  $\text{H}_3\text{BO}_3$ ,  $\text{B}_3\text{Si}$ ,  $\text{B}_6\text{O}$  and  $\text{B}_4\text{C}$ .--

BORON-BASED CONFINEMENT MATRIX FOR THE STORAGE OR  
INCINERATION OF LONG-LIFE RADIOACTIVE ELEMENTS

Field of the invention

The present invention relates to a confinement matrix for the storage of radioactive waste, composed of long-life radioactive elements such as long-life fission products and actinides. It also relates to the  
5 incineration of such elements, particularly actinides.

In used nuclear fuel reprocessing plants, some long-life actinide elements and long-life fission products remain at the end of processing, which must be conditioned with a view to long-term storage in very  
10 resistant matrices.

The materials that can be used as a matrix must have very high chemical stability, radiation stability and temperature stability characteristics to isolate the radioactive elements from the environment and keep  
15 them in this isolated state for very long periods due to their half-life value.

In the case of actinides, it is also possible to process said radioactive waste by means of transmutation in nuclear reactors, said operation being  
20 referred to in this document as "incineration". Therefore, it is also advisable to include them in matrices liable to be subjected to irradiation.

State of the related art

25 Presently, the matrix selected for the long-term storage of long-life radioactive waste is glass, but



research is ongoing to find new materials offering even better characteristics for this conditioning.

Following recent research, it was proposed to condition said waste in apatitic matrices, as disclosed  
5 in WO95/02886 [1]. Research was continued to find other materials liable to be used as a confinement or incineration matrix for long-life radioactive elements such as long-life fission products such as Cs, Sr, Tc, etc., and actinides.

10 Of the materials that could be envisaged, boron-based materials could be of interest since boron is a neutron absorbing or moderating element. To date, only one boride such as boron carbide has been used as an absorbent in fast-neutron nuclear reactor and  
15 pressurised water reactor control rods and as a moderator for incineration targets. In this way, it was observed that this material shows a high stability under irradiation due to its specific crystalline structure. However, it is subject to accelerated  
20 corrosion in aqueous media, which renders it unfit for use as a confinement matrix for long-term storage in geological formations due to the possible presence of water.

25 Description of the invention

The present invention specifically relates to the use of  $B_4C$  and other boron compounds as a confinement matrix for the long-term storage or incineration of long-life radioactive elements.

30 According to the invention, the confinement matrix for the storage or incineration of at least one long-

life radioactive element, comprises at least one crystalline boron compound of a rhombohedral structure including the long-life radioactive element(s).

In this way, the boron compounds used comprises a  
5 crystalline structure similar to that of boron carbide, which is characterised by a rhombohedral mesh which is composed, firstly, of a basic structure composed of a rigid network of polyhedrons of 12 atoms, referred to as icosahedrons, which gives the majority of the  
10 properties of said compounds and, secondly, a group of 2 or 3 atoms located in the empty spaces, i.e. at the centre of the rhombohedron. This structure is of particular interest since the atoms located in the empty spaces can be exchanged to insert radioactive  
15 element atoms while retaining the radiation stability properties of boron carbide  $B_4C$ .

According to a first embodiment of the confinement matrix according to the invention, the long-life radioactive element(s) is/are inserted in the  
20 crystalline network of the boron compound. They can thus be included in the empty spaces located at the centre of the rhombohedrons.

According to a second embodiment of the matrix according to the invention, said matrix is in the form  
25 of a composite material wherein the long-life radioactive element(s) is/are dispersed in oxide form, the standard precursor form, in the rhombohedral structured boron compound.

In this case, the boron compound may be for  
30 example  $B_4C$ ,  $B_3Si$  or  $B_6O$ .

In said second embodiment, the boron compound used corresponds to boron carbide wherein the carbon has been replaced by other elements.

5 Said replacement makes it possible to obtain the required corrosion resistance in the presence of aqueous media. Indeed, boron carbide shows said accelerated corrosion due to the formation on its surface of a layer of boric anhydride solution in aqueous media and in the presence of radiation. By  
10 replacing carbon by oxygen or silicon, the production of said boric anhydride is prevented.

Indeed, in the case of  $B_3Si$ , a film of passivating  $SiO_2$  and, in the case of  $B_6O$ , no additional oxidation into  $B_2O_3$  is possible.

15 Said matrices according to the invention also offer a high heat-resistance since they have very high melting points, of at least  $1800^\circ C$ , a good stability under irradiation, a good inertia in aqueous media and the possibility to incorporate a wide range of elements  
20 in said structure.

It was also observed that in the case of the boron compound  $B_3Si$ , said compound is subject to less degradation under irradiation by helium ions than the compound  $B_4C$ .

25 According to the invention, it is possible to adapt the composition of the boron compound to the desired use of the confinement matrix. In this way, when the confinement matrix is intended for the incineration of at least one radioactive element, it is  
30 beneficial to use a boron compound, wherein the boron

is enriched with  $^{11}\text{B}$  to benefit from the neutron moderating properties of  $^{11}\text{B}$ .

The confinement matrices according to the invention may be prepared using methods implementing powder metallurgy techniques.

In addition, the invention also relates to a method to prepare a long-life radioactive element confinement matrix comprising at least one crystalline compound of a rhombohedral structure in the crystalline network of which the long-life radioactive element(s) is/are inserted, which consists of mixing a powder of said radioactive element(s) or compound(s) of said element(s) with a boron powder or a boron precursor, and then producing a hot reaction of the powder mixture at a temperature of 800 to 1500°C and sintering the powders obtained.

In this method, the hot powder mixing reaction and sintering operations of the powders obtained may be carried out at the same time by means of reactive sintering of the powder mixture at a temperature of 1000 to 1800°C at a pressure of 30 to 200 MPa.

In this method, the powder mixture may also comprise one or more additives chosen from metals, catalysts, metal oxides or any adjuvant required to form the matrix or improve its properties.

The metals may be in particular Mg, Ca, Zn. They are used either as catalysts (for example magnesiothermic or calciothermic reaction) or as an addition of oxygen (for example ZnO).

When the powder mixture comprises a catalyst, said catalyst may be used to favour the formation of the desired rhombohedral structure boron compound.

The metal oxides used are generally also added to  
5 favour the formation of the desired boron compound. An example of oxides includes zinc oxide and magnesium oxide.

The boron precursor may be chosen from boron oxides such as  $B_2O_3$ , boric anhydride  $H_3BO_3$ , boron and  
10 silicon compounds such as  $B_3Si$ , oxygen and boron compounds such as  $B_6O$  and boron carbide  $B_4C$ .

In this method, the radioactive element may be in the form of a compound such as an oxide.

With this method, it is possible to prepare a  $B_3Si$   
15 type boron compound including in its crystalline network at least one radioactive element, using, as the powder mixture, a mixture of powders of boron, silicon and at least one radioactive element. In this case, it is possible to produce the hot reaction and sintering  
20 at the same time by carrying out reactive sintering at a temperature of 1300 to 1400°C, at a pressure of 30 to 200 MPa.

If the boron compound produced is of the  $B_6O$  type, incorporating in its network the radioactive  
25 element(s), it is possible to start with a powder mixture composed of boron powder, a metal oxide such as  $ZnO$ , and at least one radioactive element. In this case, the powders are first reacted at a temperature of 1000 to 1500°C under an inert gas stream, and the  
30 sintering is then carried out at a temperature of 1200 to 1800°C at a pressure of 30 to 200 MPa.

According to the invention, it is also possible to prepare a confinement matrix in the form of a composite material, comprising a crystalline boron compound of a rhombohedral structure wherein the long-life  
5 radioactive element is dispersed using a method comprising:

- mixing of a powder of the rhombohedral structure crystalline boron compound with a powder of the radioactive element or a compound of said element  
10 chosen from oxides, and

- pressurised sintering of the mixture obtained at a temperature of 1000 to 1800°C, at a pressure of 30 to 200 MPa.

In the latter case, the boron compound may  
15 advantageously be  $B_3Si$ ,  $B_6O$  or  $B_4C$ .

In the confinement matrix according to the invention, the radioactive element(s) included may represent 5 to 20% of the material by weight.

The invention's other characteristics and  
20 advantages will be seen more clearly upon reading the following example, which is given as an illustration and is not restrictive.

#### Detailed description of embodiment

25 The following example illustrates the preparation of a confinement matrix in the form of  $B_4C$  based composite material.

In this example,  $CeO_2$  is used to simulate  $PuO_2$  with a view to incineration matrices.

30 12 g of  $B_4C$  powder is mixed with 1.2 g of  $CeO_2$  powder, both powders having a grain size of less than

50  $\mu\text{m}$ . The homogeneous powder mixture is then subjected to sintering under a uniaxial load at a temperature of  $1800^{\circ}\text{C}$  at a pressure of 30 MPa.

This gives a composite material wherein 8% by  
5 weight of cerium is dispersed in  $\text{B}_4\text{C}$ .

Reference

[1]: WO95/02886.

CLAIMS

1. Confinement matrix for the storage or incineration of at least one long-life radioactive element, comprising at least one crystalline boron compound of a rhombohedral structure including the  
5 long-life radioactive element(s).

2. Matrix according to claim 1, wherein the long-life radioactive element(s) is/are inserted in the crystalline network of the boron compound.  
10

3. Matrix according to claim 1, wherein the long-life radioactive element(s) is/are dispersed in oxide form in the rhombohedral structured boron compound.

15 4. Matrix according to claim 3, wherein the boron compound is  $B_3Si$ .

5. Matrix according to claim 3, wherein the boron compound is  $B_6O$ .  
20

6. Matrix according to claim 3, wherein the boron compound is  $B_4C$ .

7. Confinement matrix according to any of claims 1  
25 to 6 for the incineration of at least one radioactive element, wherein the boron of the boron compound is enriched with  $^{11}B$ .



8. Method to prepare a long-life radioactive element confinement matrix comprising at least one crystalline compound of a rhombohedral structure in the crystalline network of which the long-life radioactive element(s) is/are inserted, which consists of mixing a powder of said radioactive element(s) or compound(s) of said element(s) with a boron powder or a boron precursor, and then producing a hot reaction of the powder mixture at a temperature of 800 to 1500°C and sintering the powders obtained.

9. Method according to claim 8, wherein the hot reaction and sintering are performed at the same time by means of reactive sintering at a temperature of 1000 to 1800°C, at a pressure of 30 to 200 MPa.

10. Method according to any of claims 8 and 9, wherein the powder mixture also comprises one or more additives chosen from metals, catalysts, metal oxides or the adjuvants required to form the matrix or improve its properties.

11. Method according to any of claims 8 to 10, wherein the boron precursor is chosen from  $B_2O_3$ ,  $H_3BO_3$ ,  $B_3Si$ ,  $B_6O$  and  $B_4C$ .

12. Method according to claim 9, wherein the powders of the mixture are powders of boron, a metal oxide and at least one radioactive element, wherein the reactive sintering is performed at a temperature of 1300 to 1400°C at a pressure of 30 to 200 MPa.

13. Method according to claim 8, wherein the powders of the mixture are powders of boron, a metal oxide and at least one radioactive element, wherein the  
5 powders are first reacted at a temperature of 1000 to 1500°C, under an inert gas stream, and the sintering is then carried out at a temperature of 1200 to 1800°C at a pressure of 30 to 200 MPa.

10 14. Method to prepare a confinement matrix in the form of a composite material, comprising a crystalline boron compound of a rhombohedral structure wherein the long-life radioactive element is dispersed using a method comprising:

15 - mixing of a powder of the rhombohedral structure crystalline boron compound with a powder of the radioactive element or a compound of said element chosen from oxides, and

- pressurised sintering of the mixture obtained at  
20 a temperature of 1000 to 1800°C, at a pressure of 30 to 200 MPa.

15. Method according to 'claim 14, wherein the boron compound is  $B_4C$ ,  $B_6O$  or  $B_3Si$ .

ABSTRACT OF THE DISCLOSUREBORON-BASED CONFINEMENT MATRIX FOR THE STORAGE OR  
INCINERATION OF LONG-LIFE RADIOACTIVE ELEMENTS

The invention relates to a confinement matrix for the storage or incineration of at least one long-life radioactive element, comprising at least one crystalline boron compound of a rhombohedral structure including the long-life radioactive element(s).

The boron compound may be of the  $B_3Si$ ,  $B_6O$  or  $B_4C$  type.

B 13403.3 MDT

*Declaration, Power Of Attorney and Petition*

Page 1 of 3

WE (I) the undersigned inventor(s), hereby declare(s) that :

My residence, post office address and citizenship are as stated below next to my name,

We (I) believe that we are (I am) the original, first, and joint (sole) inventor(s) of the subject matter which is claimed and for which a patent is sought on the invention entitled

BORON-BASED CONFINEMENT MATRIX FOR THE STORAGE OR INCINERATION OF LONG-LIFE  
RADIOACTIVE ELEMENTS

the specification of which

☐ is attached hereto.

☒ was filed on April 12, 2002

as Application Serial No. 10/089,099

and amended on

☒ was filed as PCT international application

Number PCT/FR00/02841

on October 12, 2000

and was amended under PCT Article 19

on

We (I) hereby state that we (I) have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

We (I) acknowledge the duty to disclose information known to be material to the patentability of this application as defined in Section 1.56 of Title 37 Code of Federal Regulations.

We (I) hereby claim foreign priority benefits under 35 U.S.C. § 119 (a)-(d) or § 365 (b) of any foreign application(s) for patent or inventor's certificate, or § 365 (a) of any PCT International application which designated at least one country other than the United States, listed below and have also identified below, by checking the box, any foreign application for patent or inventor's certificate, or PCT International application having a filing date before that of the application on which priority is claimed. Prior Foreign Application (s)

Application No.	Country	Day/month/Year	Priority Claimed
99 12766	FRANCE	13 OCTOBER 1999	<input checked="" type="checkbox"/> YES <input type="checkbox"/> NO
_____	_____	_____	<input type="checkbox"/> YES <input type="checkbox"/> NO
_____	_____	_____	<input type="checkbox"/> YES <input type="checkbox"/> NO
_____	_____	_____	<input type="checkbox"/> YES <input type="checkbox"/> NO

We (I) hereby claim the benefit under Title 35, United States Code, § 119 (e) of any United States provisional application(s) listed below.

(Application Number)

(Filing Date)

(Application Number)

(Filing Date)

We (I) hereby claim the benefit under 35 U.S.C. §120 of any United States application(s), or § 365(c) of any PCT International application designating the United States, listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States or PCT International application in the manner provided by the first paragraph of 35 U.S.C. § 112, I acknowledge the duty to disclose information which is material to patentability as defined in 37 CFR § 1.56 which became available between the filing date of prior application and the national or PCT International filing date of this application.

Application Serial No.

Filing Date

Status (pending, patented,  
abandoned)

And we (I) hereby appoint : Michael N. MELLER, Registration Number 20,779; Eugene LIEBERSTEIN, Registration Number 24645, our (my) attorneys, with full powers of substitution and revocation, to prosecute this application and to transact all business in the Patent Office connected therewith; and we (I) hereby request that all correspondence regarding this application be sent to the firm of ANDERSON KILL & OLICK, P.C. whose Address is . 1251 Avenue of the Americas, New York NY 10020-1182

We (I) declare that all statements made herein of our (my) own knowledge are true and that all statements made on information and belief are believed to be true ; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such wilful false statements may jeopardise the validity of the application or any patent issuing thereon.

DEUTSCH Sylvain

NAME OF FIRST SOLE INVENTOR

Signature of Inventor

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Citizen of : FRANCE

Post Office Address : The same as residence

April 24, 2002

Date \_\_\_\_\_

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 Signature of Inventor  
 April 24, 2002  
 Date

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 NAME OF THIRD INVENTOR  
 Signature of Inventor  
 April 24, 2002  
 Date

4W SIMEONE David  
 NAME OF FOURTH INVENTOR  
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 April 24, 2002  
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 Post Office Address : The same as residence

Residence :

Citizen of :

Post Office Address : The same as residence